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(19)(CA) **APPLICATION FOR CANADIAN PATENT** (12)

(54) Method and Apparatus for Environmentally-Friendly Mass De-Acidification of Books and Other Paper Products

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2009621

Abstract:

Method and apparatus for environmentally-friendly mass de-acidification of books and other printed and paper products employing pre-drying by high frequency radiation in a vacuum, a neutralization phase employing de-acidification solutions and evaporation of the solvent used also by high frequency radiation in a vacuum, whereby, preferably, a single treatment chamber (1) is utilized for pre-drying, neutralization and drying-up, whose inner cavity can be exposed to the HF/microwave radiation of a generator (2), such chamber being connected to the devices (4, 8, 9; 10 to 19 and 5) that connect to chamber (1) inside a closed, movable enclosure for the purpose of supplying and draining off the solutions used, for evacuation and for separation of the vapours present in the spent air given off during the drying phase, thus removing the threat to the environment. The proposed method and the apparatus permit rapid, economical and environmentally-safe mass de-acidification with practically total solvent recovery.

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Method and apparatus for environmentally-friendly mass de-acidification of books and other paper products

The present invention relates to an environmentally safe method for simultaneously de-acidifying large quantities of books and many other types of printed material such as magazines, documents etc. The present invention relates furthermore to an apparatus capable of implementing the proposed method.

The disadvantages attending the storage of printed material and paper products, a notable example of which are books, entails progressive damage of the paper itself, largely due to the liberation of trace amounts of acids in the paper. Such damage, left unaddressed, may cause, after a few decades, the complete disintegration of the stored paper.

Preservation of the hundreds of millions of documents held worldwide in archives and libraries would require the neutralization of the acids present in the paper and the impregnation of the paper with a sufficient amount of a substance that would act to preclude future acid liberation in the paper.

In view of the enormous quantities of books and other documents that would have to be treated, only those methods permitting treatment of the entire book would be suitable, which is to say methods not requiring unravelling the book binding and treating each page individually. The same considerations, which likewise necessitate the treatment of bound or otherwise collected paper pages, also apply to all types of archival holdings.

One prior art method involves treating books with the fumes of metal alkyls or, more particularly, those of diethyl zinc. The metal alkyls are transformed, by means of the moisture present in the paper, into the oxides of the metals, (e.g. zinc oxide), which, remaining in the paper, serve to effectively neutralize the liberated acids. The metal alkyls used in this role, being spontaneously combustible in the presence of air, pose a constant fire and explosion hazard while being handled, and accordingly require extra care in handling as well as appropriate labelling.

Another prior art method involves treating books after drying with a solution of a magnesium organic compound, an example of which can be methyl magnesium carbonate in a suitable solvent. During this procedure, the magnesium compound is transformed by the moisture contained in the paper into magnesium oxide and magnesium carbonate, both of which are capable of neutralizing acids. Fluorocarbons are especially suitable as solvents, an example of which is trichlorotrifluorethane mixed with an alcohol such as methanol.

In addition to the advantages of non-combustibility and non-toxicity, fluorocarbons and chlorofluorocarbons are also highly compatible with most book materials such as paper, cardboard, printing inks, glue and other adhesives, and are thus especially well suited for the purpose under consideration.

Besides their high price, one distinct disadvantage of the above-mentioned chemical substances is that their escape into the atmosphere poses a danger to the environment.

Consequently, existing environmental legislation requires the recovery of a large percentage of such substances and the thorough purification of the air discharged from the treatment enclosure.

Consequently, books and other printed matter must, following treatment, be dried in such a way that practically all of the solvent is removed. Furthermore, the solvent-laden air produced by drying must undergo a purification process that serves to remove virtually all of the solvents employed.

While the drying of solvent-containing bulk books is not known to entail major problems, the drying of compact, bound paper has, until now, necessitated very long drying cycles. Although book drying cycles can be significantly reduced by lowering the ambient pressure, the reduced pressure unfortunately hampers the supply of heat necessary for solvent evaporation.

To the reduced pressure drying process (i.e. vacuum drying) was added a method employing heat radiation for introducing into the objects to be dried the heat required for evaporation. This method, when used to treat books, is effective only at very low heat; otherwise the books, and especially the book sizing, risk damage.

The present invention relates to a method for mass de-acidification of books and other paper products which, while permitting rapid and thus economical de-acidification of books and other products does not harm the environment. Furthermore, a device suitable for implementing the proposed method is also disclosed.

Thus, one embodiment of this invention comprises a method suitable for the environmentally-friendly mass de-acidification of books and other paper products employing a neutralization process using de-acidification solutions and subsequent evaporation of the solvent, characterized in that the books or other paper products are pre-dried in vacuum by heating with high-frequency radiation and said books or other paper products are dried following de-acidification in a

vacuum by heating with high-frequency radiation.

A further embodiment comprises an apparatus for carrying out the above method. Such apparatus comprises a single treatment chamber having an inner cavity, a high frequency radiation generator for directing radiation into said inner cavity, means for feeding de-acidification solution into said inner cavity, means for drawing off solution, suction means for pre-drying the cavity and for drawing off spent air and solvent vapour after solvent removal, and means for separating the solvent vapour from the spent air.

The discovery that solvents effective in the treatment of books, such as alcohols, fluorocarbons and chlorofluorocarbons are well adapted to high-frequency heating, was quite surprising. Dehydration by high frequency heating was until the present considered only in other areas of drying technology. The physical and chemical properties of water suit it well to high frequency or microwave radiation heating, by means of which it is evaporated.

Successful application of high frequency heat not only to the above-mentioned solvents but also to other suitable solvents, is not only novel and unexpected, but affords important advantages. In comparison experiments, it was possible to reduce preliminary drying cycles to less than a fiftieth of those required for the process wherein printed matter is safely heated to a moderate level inside a vacuum. A considerable time saving was also achieved in the solvent evaporation step which, with conventional methods applied in the presence of normal or reduced pressure, was able to remove virtually all of the solvent present in the paper.

Besides being otherwise advantageous, the speed with which the proposed method is executed at last opens the way to large-scale de-acidification of bulk printed matter, a need that will be acutely felt in the near future. The drying step, although efficient, does not harm the treated material, due to the employment of low heat levels. Rapid, virtually complete solvent removal also permits use of effective, yet environmentally-harmful solvents.

The speed of application of the proposed method moreover permits operation of preliminary drying, neutralization and solvent evaporation phases inside a single chamber, since time-consuming preliminary drying procedures in special driers are obviated. Use of a single chamber, moreover, permits the operation of a closed system, which, if effectively utilized, permits the complete capture and controlled processing of the spent air. Furthermore, the expensive solvent used is recovered by a separation step that reduces damage to the environment by retrieving all of the solvent laden air expelled by the process. Essential treatment substances, waste air purification substances and their storage tanks are arranged in a compact, mobile apparatus that can be transported directly to the storage site, i.e. to the archive or library. The proposed system, being self-supporting, can be operated by library personnel who are able to control process functions by manipulating a number of automatic controls. Irradiation with either HF or microwave radiation does not require high-technology hardware. The same high frequency or microwave radiation can be employed for both pre-drying and subsequent evaporation, thus permitting use of a dual-chamber device comprising a separate pre-drying chamber, enabling at once the pre-drying and subsequent treatment of bulk quantities of books.

The proposed method and apparatus for its implementation are also disclosed in greater detail with the aid of figures, as follows:

Fig. 1 shows a first embodiment example of the proposed apparatus; and

Fig. 2 shows a second, modified version of the proposed apparatus.

The apparatus in Fig 1 has as its chief component a single chamber 1 employed for pre-drying, treatment with chemical solvents, and final drying. Treatment chamber 1 is also equipped with an HF or microwave generator 2 by means of which, for example, the chamber interior can be exposed to high frequency radiation of 27.12 MHz or microwave radiation

of 2450 MHz as is indicated schematically. The possibility of generating microwave radiation or HF radiation in the kind of cylindrical cavity used in the present example is known in other technical art. In the present embodiment example, a microwave chamber was exposed to a radiation frequency of 2450 MHz.

Chamber 1 connects via a valve to an air dryer 3 and is thus supplied with dried air for flushing. Chamber 1 is further connected via valves and purification filters that are provided in the inlet and outlet lines, respectively, to a reservoir 4 containing treatment solution. A feed pump is provided upstream of the filter in the inlet line.

Chamber 1, moreover, connects to a vacuum pump 5 for whose efficient operation the chamber pressure is measured and controlled by a pressure sensor PC (connected to a valve for flushing air as indicated by the broken line). The temperature in the chamber can also be controlled by a temperature probe TC with corresponding feedback to generator 2. The temperature of the outer wall of the chamber can be modified by direct or indirect electrical heating of the jacket, indicated by 6, so as to avoid water vapour condensation on the chamber wall and to accelerate microwave drying.

A compressor 7, connected via a valve to vacuum pump 5, connects to chamber 1 and constitutes the connection to the solvent preparation and recovery devices. A solvent tank 8 is also connected to the latter devices via the corresponding recovery line. Both tank 8 -- a storage tank containing the active process compound -- in the present case methylmagnesium carbonate -- and a supply tank, are supplied from reservoir 4 via supply lines.

Solvent tank 8 is fed from a device 10 that processes solvents by drying and neutralization and that connects to a solvent collecting tank 11. The latter is fed from a condenser 13 connected to a refrigeration unit 12. Condenser 13, supplied via compressor 7 with spent air given off during the final drying step, connects to a device 14 for absorptive

spent-air purification via a valve that is also connected to a blower 15 that completes an absorption-separation loop including device 14 and an air cooler 16 that is connected to both device 14 and blower 15. The former is fed with desorption steam and, depending on the setting of the valve located between condenser 13 and blower 15, with either the spent air from the compressor or, via the blower, with additional air for dilution of the spent air. Spent air dilution is effected if a valve, located between air cooler 16 and blower 15, is set to introduce additional air. Furthermore, a spent air valve serving to recirculate purified spent air is provided upstream of the air cooler.

Device 14, which purifies spent air by absorption, also feeds regenerated solvent to solvent collector 11 via an additional condenser 17 and a device 18 for water/desorptive phase separation that connects to water purifier 19.

The single-chamber method of environmentally-friendly mass de-acidification of books as implemented by employing the apparatus illustrated in Fig. 1, operates thusly: A quantity of books is deposited for processing, their backs facing downward, in baskets made of a suitable material, which may be, for example, polyethylene or polypropylene. Insertion of spacers helps immobilize such books in orderly fashion inside the baskets.

The books so arranged are then pre-dried in vacuum-and-pressure proof chamber 1, which is, for the purpose of this procedure, evacuated by means of a vacuum pump 5 to a pressure of about 100 mbar. While being pre-dried, the books are heated to a maximum of 60°C in the microwave field which requires the engagement of the already-mentioned pressure and temperature controls. Air dryer 3, by circulating a gentle air current through the chamber together with the simultaneous heating of chamber jacket (6), serves to eliminate water condensation on the chamber wall and thus accelerates drying. The spent air is, during this pre-drying phase, pumped from the vacuum to the outside air via pump 5.

As soon as the drying step has reduced the residual

moisture in the books to a predetermined value, vacuum pump 5 is turned off and treatment chamber 1 is flooded with neutralization solution. Solution impregnation is improved if an overpressure is created in the chamber by using the feed pump located in the supply line leading from tank 4. Impregnation may, if required, be enhanced by pressure pulsation and/or acoustic bombardment at a suitable frequency.

When the required reaction time has elapsed, the resulting solution is removed to storage tank 4, and simultaneously relieved of dirt particles and washed off magnesium oxide by a fine filter located in the line. Next, microwave generator 2 is switched on again to commence evaporation of solvent.

The solvent vapours generated by heating the books are suctioned off first by means of refrigeration compressor 7 and toward the end of the drying phase by means of vacuum pump 5 which is switched on by means of a valve. The vapours are cooled down and concentrated inside condenser 13. A considerable portion of the suctioned solvent vapours are condensed at this point and then piped into solvent collecting tank 11.

Devices 14, 15 and 16, which form an absorptive waste-air purification loop, continue removal of the solvent vapours until the legal tolerance values have been attained. Introduction of additional air into such loop via blower 15, further reduces the proportion of solvent in the expelled spent air.

After the maximum amount of solvent has been removed in chamber 14, the absorption process is restarted by the introduction of steam and if necessary, creation of a vacuum. After condensation (17) and phase separation (18), the solvent is gathered in reservoir 11, combined with the separated-out solvent pumped from condenser 13, neutralized and dried in device 10, and finally piped to solvent tank 8.

After separation of the methanol added in the water purification device 19, the water condensate is either used again or channeled into the waste water stream.

Rigorous adherence to legal emission standards is ensured by the routing to the spent air purifier of all spent air, in addition to the solvent collected from the dryer, i.e. spent air collected from the chamber during filling and emptying, spent air from the storage tanks, and spent air from the water purification device.

The apparatus shown in Fig. 2 corresponds to the device of Fig. 1 save for the addition of an auxiliary pre-drying chamber 20 which, also connecting via a valve to air dryer 3, is exposed to microwave or HF radiation supplied by generator 2. This second version of the system operates analogously to that of Fig. 1, whereby however a higher throughput capacity is enabled due to an arrangement permitting concurrent pre-drying of one load of books and the deacidification of a second, already pre-dried load. The system is also completely closed, since spent air processing in chamber 1 takes place, as in the example described, without any emission of solvents into the environment. In addition to the dual-chamber method already disclosed, the utilization of two treatment chambers and one pre-drying chamber, is theoretically possible, since pre-drying can, in general, be carried out in shorter periods of time than the final drying phase.

The following experimental results were obtained using the method described. Books having a moisture content of between 8 and 10% were pre-dried, under a pressure of 50 mbar and at a high frequency power of 500 watts and then exposed to a radiation frequency of 2450 MHz. This method reduced moisture content in the paper to 2% in only 30 minutes. By way of contrast, employment of vacuum drying without high frequency heating requires from 30 to 40 hours to reduce the residual moisture content to 1 or 2%.

A final drying of books that have soaked up 100 to 120% of their dry weight of the solvent trichlorotrifluorethane after neutralization treatment, uses the same high frequency radiation and 280 watts of high frequency power. After 15 minutes, this procedure yielded solvent-free books whose temperature, upon termination of drying, was about 60°C.

24 to 30 hours are required to dry out books when stored in ca. 20°C air. This method in addition to not achieving such effective removal of solvent, poses a certain degree of environmental hazard. Also, vacuum drying at a terminal pressure of 1 mbar and at a temperature of 50°C, required 2 to 5 hours with conventional heating means.

Claims:

1. Method suitable for the environmentally-friendly mass de-acidification of books and other paper products employing a neutralization process using de-acidification solutions and subsequent evaporation of the solvent,

characterized in that the books or other paper products are pre-dried in vacuum by heating with high-frequency radiation and said books or other paper products are dried following de-acidification in a vacuum by heating with high-frequency radiation.

2. Method according to Claim 1, wherein solutions of a magnesium organic compound in methanol, ethanol, fluorocarbon, chlorofluorocarbon, benzene or a mixture of such substances are employed for de-acidification, and wherein the pre-drying, de-acidification and evaporation of solvent are carried out in a treatment chamber.

3. Method in accordance with Claim 1, wherein the vapours, or the solvent removed from the spent air released during the drying stage, are separated by means of condensation and/or adsorption and/or absorption.

4. Method in accordance with Claim 1 or 3, wherein pre-drying is carried out in a separate chamber.

5. Method in accordance with Claim 1, 2 or 3, wherein high frequency radiation of 27.12 MHz is used in the heating step.

6. Method in accordance with Claim 1, 2 or 3, wherein high frequency radiation of 2450 MHz is used in the heating step.

7. Apparatus for the mass de-acidification of books and other paper products in which the books and other paper products are neutralized with de-acidification solutions and the solvents are subsequently evaporated,

said apparatus comprising a single treatment chamber

having an inner cavity, a high frequency radiation generator for directing radiation into said inner cavity, means for feeding de-acidification solution into said inner cavity, means for drawing off solution, suction means for pre-drying the cavity and for drawing off spent air and solvent vapour after solvent removal, and means for separating the solvent vapour from the spent air.

8. An apparatus according to Claim 7, which includes a second chamber for vacuum pre-drying and a high frequency radiation generator for directing radiation into said second chamber.

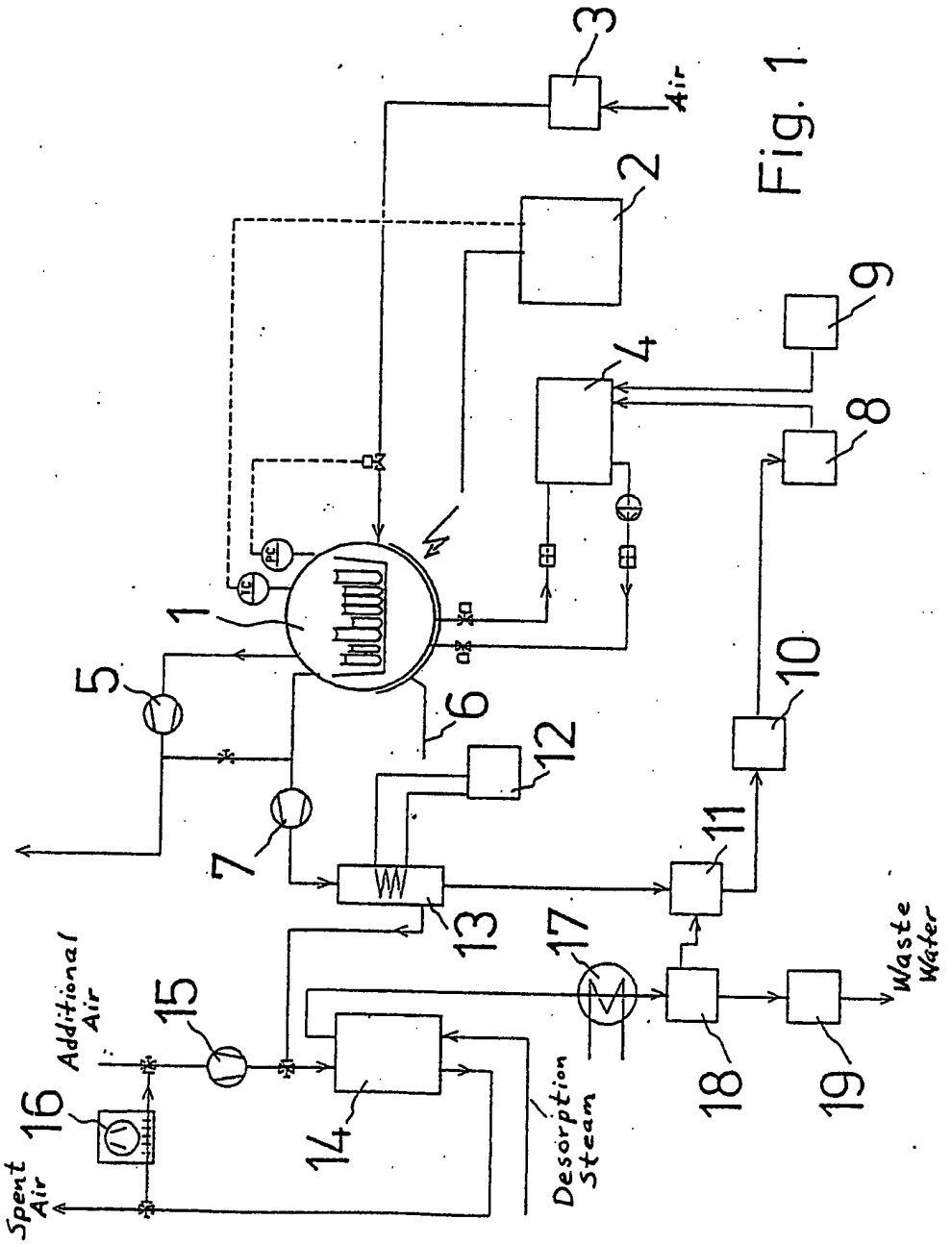

Fig. 1

Kirby, Eades, Gale, Baker & Power

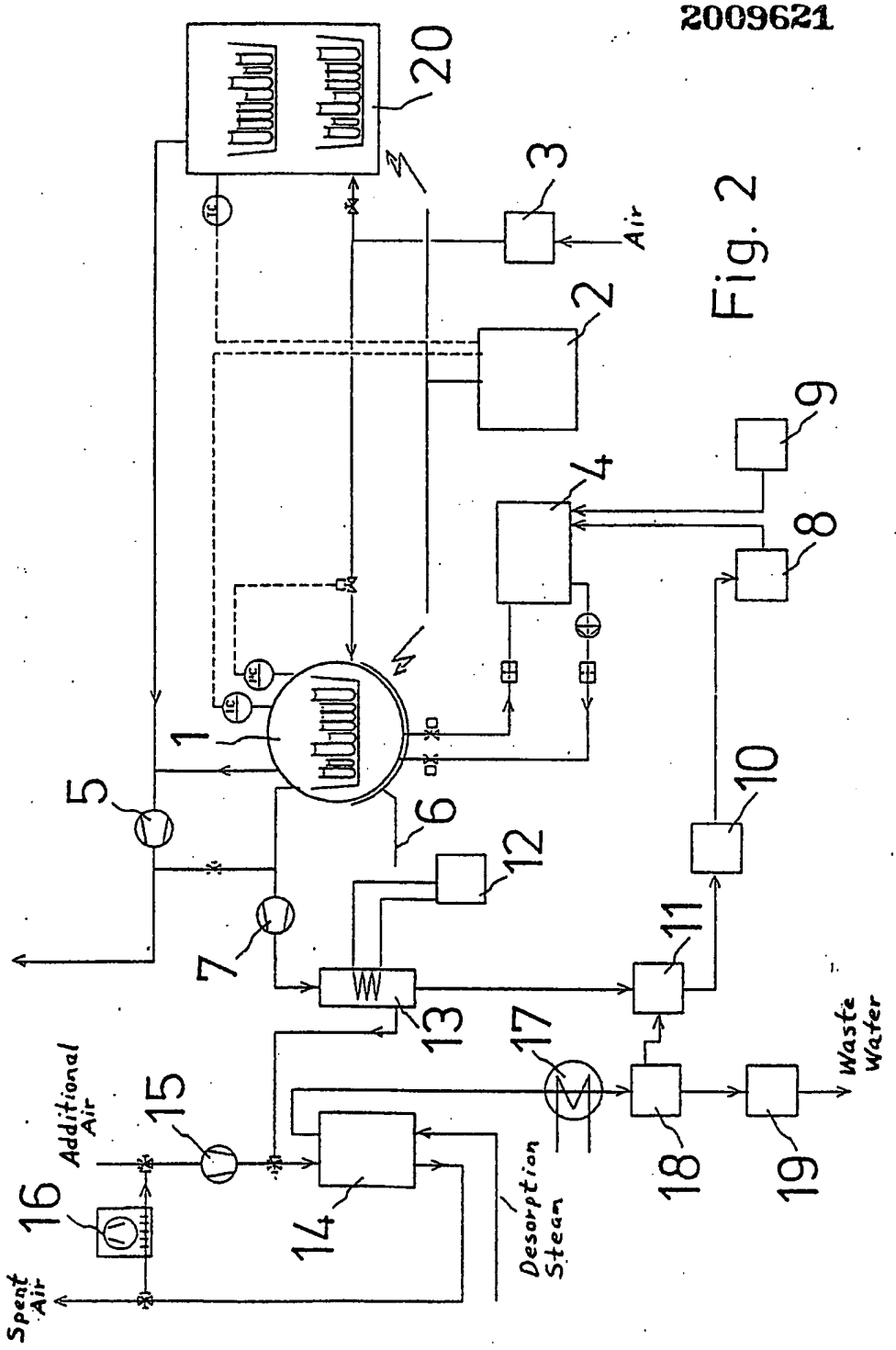

Fig. 2